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STRUCTURE AND SURFACE CHARACTERIZATIONS OF HIGH
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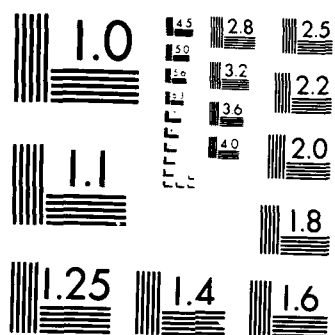
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STRUCTURE AND SURFACE CHARACTERIZATIONS OF HIGH STRENGTH CARBON FIBERS

SIN SHONG LIN

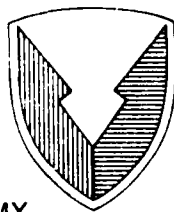
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ABSTRACT

→ Six commercial carbon fibers are characterized by X-ray diffraction, scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS), or Electron Spectroscopy for Chemical Analysis (ESCA). Physical, structural, and surface parameters required to describe major properties of carbon fibers are examined and discussed. The correlation of these parameters is essential for developing optimizing and desired carbon composite materials.

Key words:

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INTRODUCTION

Fiber reinforced composites are widely used in high technology design and manufacture of helicopters, aircraft, missiles, and other weapon components. The toughness of the designed composite depends on the properties of the fibers, bulk matrix, and the interfaces between them. Understanding of the fiber-matrix interfaces and the correlation of these findings to the mechanical properties of the composite is essential for developing micro-mechanical models and failure mechanisms of the composite, and might lead to improvement and optimization of desired composite materials.

The structures of carbon fibers (CF) are fairly well documented,¹⁻⁴ but their surfaces after liquid oxidation modification and oxygen gas treatment have not been extensively investigated. The fibers are usually given a surface treatment after fabrication to improve adhesive properties⁵ of the surface. Understanding of the interactions between fiber and matrix (e.g., the chemical nature of fiber surface layers, interactive forces between fiber and matrix, and fiber surface roughness and porosity) is essential for a complete structural and surface characterization.

The present investigation was carried out as part of The Technical Cooperative Program (TTCP) in which England, Canada, and the U.S. participated. Using available analytical facilities in the Materials Characterization Division of the U.S. Army Materials Technology Laboratory, the structure and chemistry of fibers were examined in detail. The data obtained was used to classify and grade various commercial fibers, and might be helpful in designing tougher composite materials. The current study is aimed at selecting physical and chemical parameters sufficient to describe major characteristics of the fibers that can be employed on a selection basis in order to optimize properties of the fabricated composite material. They include: (1) major mechanical properties, (2) crystal structure information, and (3) morphological chemical characteristics of the fiber surface.

EXPERIMENTAL

Fiber Samples

The following carbon fiber samples, obtained from the TTCP program, were made by Courtaulds Co. and distributed by Royal Aircraft Establishment (RAE):

- (A1) Grafil Apollo XA-U HS 12,000 filament tow. Reference 6662-1, Batch No. 5257, Strength 5.10 GPa, Modulus 254 GPa, Density 1.82 g/cc, Weight per unit length 396 mg/m.
- (A2) Same as A1, Batch No. 5258, treated and unsized.
- (A3) Same as A1, Batch No. 5256, treated and 1% size A.

1. DELMONTE, J. *Technology of Carbon and Graphite Composites*. Chapter 6, Van Nostrand Reinhold Co., 1980.
2. SCOLA, D.A. *Composite Materials*. E.P. Plueddemann, ed., Chapter 7, v. 6, Academic Press, 1974.
3. SCOLA, D.A., and BROOKS, C.S. *Surface Aspects of New Fibers, Boron, Silicon Carbide, and Graphite*. *J. Adhesion*, v. 2, 1970, p. 213-237.
4. RIGGS, D.M., SHUFORD, R.J., and LEWIS, R.W. *Graphite Fibers and Composites*. Handbook of Composites, G. Lubin, ed., Van Nostrand Reinhold Co., 1982.
5. DRAZEL, L.T. *The Surface Compositions and Energetics of Type A Graphite Fibers*. *Carbon*, v. 15, 1977, p. 129-138.

- (B1) Grafil Apollo IM 12,000 filament tow. Reference 6661-1, Batch No. 5447, Strength 3.90 GPa, Modulus 306 GPa, Density 1.76 g/cc, Weight 370 mg/m.
- (B2) Same as B1, Batch No. 5445, Strength 3.82 GPa, Modulus 308 GPa, treated and unsized.
- (B3) Same as B1, Batch No. 5443, Strength 3.90 GPa, Modulus 313 GPa, treated and 1% size A.

Instruments/Procedures

X-Ray Powder Diffraction

Norelco X-ray power supply and diffractometer units were employed to characterize structure parameters. The fiber was first ground into powder by a SPEX miller, and the X-ray powder diffraction pattern was taken using $\text{Cu K}\alpha$ radiation on a strip chart recorder. The patterns were then transferred to an IBM personal computer, and the structure parameters were computed.

Scanning Electron Microscopy (SEM)

A JEOL JXA-840 scanning electron microanalyzer was used to examine surface morphology of the fibers. The irregularities of the fibrous surface could be examined in detail under high magnification. The cross-sectional morphology of the fiber could reveal the internal array of graphite basal planes.

ESCA Surface Study

The ESCA/Auger instrument made by Physical Electronic Industries, Inc., PHI Model 548 with a double pass cylindrical mirror analyzer (CMA) and $\text{MgK}\alpha$ X-ray radiation, was used for surface characterization. To prepare the specimen, a bundle of fibers was densely packed and aligned to give an area of $\sim 1/2$ -inch square. The fiber ends were trimmed and clamped with a conductive, adhesive copper tape.

Structural and Surface Properties

The following information, observed or calculated from X-ray diffraction, ESCA, and SEM experiments, is essential for a thorough characterization of the fiber:

1. X-Ray Diffraction

- (a) Average graphite layer spacing (from the 002 peak position)
- (b) Average crystal size L_c (from the 002 peak width)
- (c) Average crystal size L_a (from the 100 peak width)
- (d) Average lattice dimension a-axis (from the 100 peak position)
- (e) The ratio of the peak area to the diffused area
- (f) The ratio of the 002 peak area to the total diffraction area
- (g) The ratio of the 100 peak area to the total diffraction area
- (h) The ratio between the 100 and 002 peak areas
- (i) Crystallinity index (from a comparison of the X-ray diffraction patterns of known crystallized and amorphous carbons).

2. SEM Morphology Studies

- (a) Morphological examination of surfaces along the axis of carbon fibers and fiber cross-sections
- (b) Average diameters of carbon fiber samples.

3. ESCA Surface Studies

- (a) Oxygen-to-carbon peak area ratio (calculated from the integrated area under the O_{1s} and C_{1s} peaks in ESCA spectra)
- (b) Oxidized carbon-to-carbon ratio (the ratio of oxygen attached to carbons, including hydroxyl, ether, ester, carbonyl and carboxyl functional groups, with no oxygen linkage from the C_{1s} peak profile in ESCA spectra).

RESULTS

The experimentally obtained parameters listed above together with the mechanical properties supplied by manufacturers are shown in Table 1. The procedures for obtaining these parameters are well documented,⁶⁻⁸ and brief descriptions are given below.

Table 1. CHARACTERISTICS OF CARBON FIBERS

Parameter	A-1	A-2	A-3	B-1	B-2	B-3
1. U.T. Strength (GPA)	5.1	5.1	5.1	3.9	3.82	3.9
2. Modulus (GPA)	254	254	254	306	308	313
3. Max. Strain (%)						
4. Density (g/cc)	1.82	1.82	1.82	1.76	1.76	1.76
5. Diameter (μm)	4.5	4.5	4.5	4.8	4.8	4.8
6. Surface Area (m^2/g)						
7. d-002 Spacing (\AA)	3.55	3.56	3.57	3.50	3.49	3.51
8. Dimension L_c (\AA)	15.6	15.1	17.0	28.1	23.7	25.0
9. Dimension L_a (\AA)	79.3	66.9	62.1	77.3	91.2	87.8
10. Frac. 002/Total	0.397	0.341	0.312	0.348	0.376	0.373
11. Frac. 100/Total	0.031	0.032	0.039	0.050	0.032	0.033
12. Frac. Peak/Total	0.485	0.418	0.415	0.442	0.473	0.482
13. Ratio 100/002	0.079	0.093	0.124	0.143	0.086	0.089
14. Frac. Surface Oxygen	0.051	0.14	0.15	0.033	0.092	0.161
15. Surface C-O/C Ratio	0.184	0.281	0.284	0.221	0.319	0.449
16. Crystalline Index	0.35	0.32	0.28	0.43	0.48	0.46

The physical parameters from lines 1 to 6 are obtained from the literature provided with commercial fibers; occasionally, some are missing. Average diameter (No. 5) and surface area (No. 6) can be estimated from the SEM photographs. The surface area could also be approximated from the average diameter and surface roughness.

6. HECKMAN, F.A. *Microstructure of Carbon Black* Rubber Chemistry and Technol., v. 37, no. 5, December 1964.

7. KOZLOWSKI, C., and SHERWOOD, P.M.A. *X-Ray Photoelectron Spectroscopic Studies of Carbon Fiber Surfaces. Part VII, Electrochemical Treatment in Ammonium Salt Electrolytes* Carbon, v. 24, 1986, p. 357-363.

8. HOPFGARTEN, F. *ESCA Studies of Carbon and Oxygen in Carbon Fibers* Fiber Sci. Technol., v. 12, 1979, p. 283.

However, due to irregularities on the fiber surface, reliable results can be obtained only from gas absorption measurement.

The surface morphologies observed from the SEM images of the untreated, treated, and treated-sized Grafil XA-UHS carbon fibers revealed many distinct appearances as shown in Figure 1. Under 3000X magnification, all surfaces have fine striations along the length of the fiber. The untreated carbon fibers are rather smooth, without scales, deposits or pits; however, the treated and treated-sized fibers show many coagulations, scales and wrinkles. Moreover, tiny chips and debris from cutting had adhered to the surfaces. Occasionally a layer of coating or scale can be seen on the cross sections of the sized filaments. The internal array of graphite basal planes appears to be random from other magnified SEM images (not shown).

The parameters listed from lines 7 to 13 are obtained from X-ray diffraction data. A typical X-ray diffraction pattern is shown in Figure 2. The d-002 spacing (No. 7) is calculated from the position at the maximum peak height. The maximum position is determined by the mid-point of the peak width at half-height. The lattice spacing is calculated from the Bragg equation as:

$$n\lambda = 2d \sin\theta \quad (1)$$

where

λ = wavelength of X-rays,
 d = distance between planes of the same Miller indices, and
 θ = angle of diffraction.

The dimensions L_c and L_a (Nos. 8 and 9) of graphite crystallites are calculated from the half-widths of the 002 and 100 peaks. The Scherrer equation,

$$L = K\lambda/B \cos\theta, \quad (2)$$

where

L = crystallite dimension,
 B = half-width in radians,
 θ = diffraction angle,
 λ = wavelength, and
 K = shape factor,

is used for the computation. The constant K is taken as $K_a = 1.84$ for L_a and $K_c = 0.90$ for L_c . A background correction is made before the half-width B calculation. Since the 100 peak is usually overlapped with the 110 peak, only the left shoulder and the maximum position of the peak are used to establish the half-width; this might introduce a large uncertainty in the L_a calculation. It is well known that the a -axial unit cell length is fairly uniform among many graphite-like structures. Consequently, the position of the 100 peak is assumed to be identical for all diffraction patterns.

The fractions of peak areas 002 and 100 to the total diffraction area (Nos. 10 and 11, Table 1) are calculated from the X-ray diffraction pattern. The background diffraction is obtained from a blank sample holder with only Scotch mounting tape. These two parameters denote the proportions of crystalline carbons oriented along

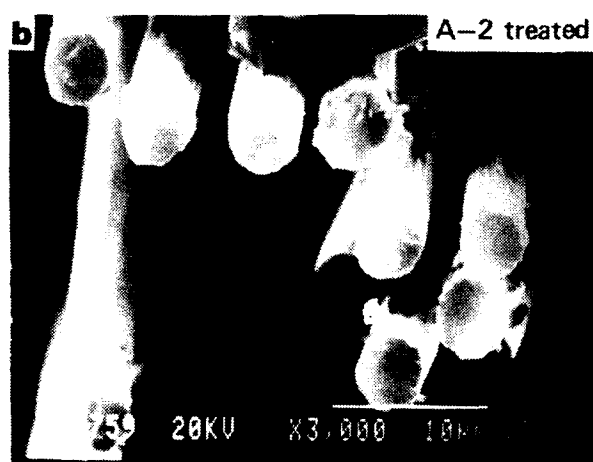
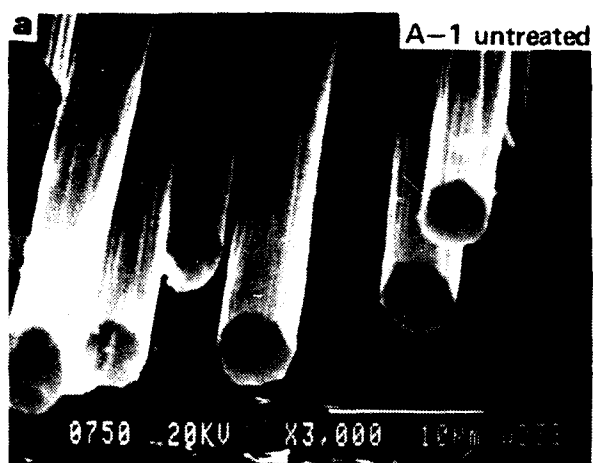


Figure 1. SEM images of carbon fibers.

the two major axes (a and c). The orientation of the graphite crystallites is an important factor in determining the physical strength of a continuous carbon filament.

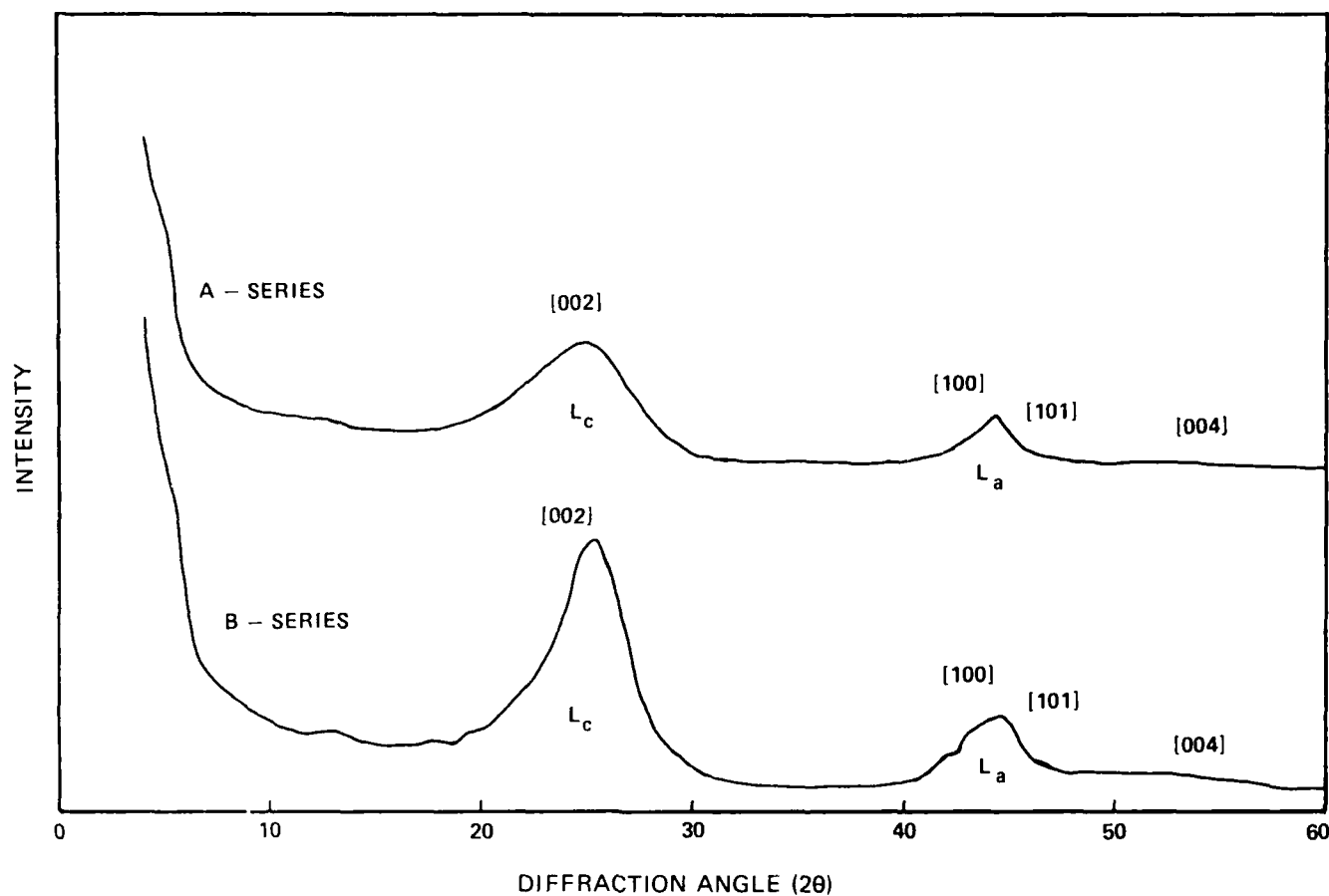


Figure 2. A typical X-ray diffraction pattern of carbon fiber. Two diffused broad peaks are usually observed in the range 4-60° under Cu radiation. The d-002 spacing is calculated from the peak position determined by half-widths, and crystallite dimensions L_a and L_c from respective peak widths at given Miller indices. Other parameters listed in Table 1 are also computed from the areas of diffraction. B-series fibers have larger sizes of crystallites than A-series fibers.

Since the physical characteristics of fibers are, in some cases, dependent on the crystalline and amorphous portions of the carbon structures, the fraction of all peak areas to the total diffraction area (No. 12) is used to demonstrate the proportion of carbons in crystalline form. This parameter approximates the degree of crystallinity of the fiber.

The ratio of two peak areas (100 and 002, No. 13) illustrates the orientation of crystallites in the fiber structure. While this ratio may duplicate the preceding parameters, a clear crystallite arrangement can be easily derived from this variable.

The two surface parameters derived from ESCA peaks, shown in Figure 3, are used to demonstrate surface atomic constituents of the fiber. The adhesion behavior of the fiber surface is strongly dependent on the amount of oxygen on the fiber surface.^{5,8,9} Thus, the concentration of oxygen on the surface, regardless of the chemical form or the amount of oxygen attaching to carbon (Nos. 14 and 15), is intrinsic to surface adhesion. Parameter 14 is calculated from the relative amount

9. ISHITANI, A. *Application of X-Ray Photoelectron Spectroscopy to Surface Analysis of Carbon Fiber*. Carbon, v. 19, 1981, p. 269.

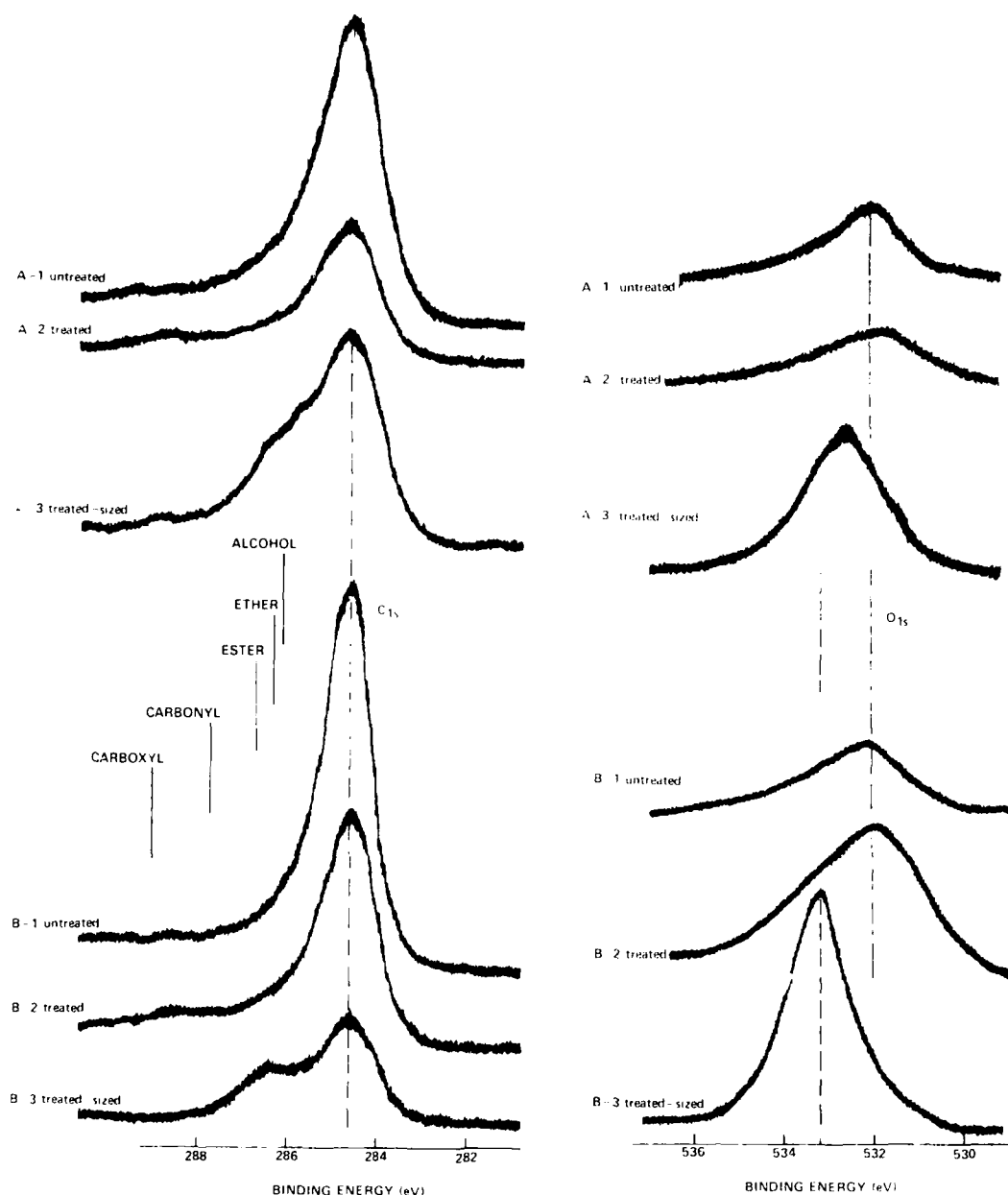


Figure 3. Carbon and oxygen peak profiles in ESCA spectra. In carbon profiles, the oxygen content increases with the treated and the treated-sized fibers as shown on the shoulders of these peak profiles. These oxygen are found to be alcohol, carbonyl, lactone and carboxyl function groups. In oxygen profiles, the treated fibers show wide peak half-widths, indicating there are many different binding states of oxygen on the modified surface.

of oxygen on the surface. First, the areas under all peaks are integrated and corrected for atomic sensitivities. Then, the atomic fraction of oxygen is derived from the integrated and corrected concentrations of surface atoms, including carbon, oxygen, and nitrogen. The second parameter (No. 15) is the ratio of oxygen-attached carbon to unreacted carbon that may show some active oxygen-carbon linkages on the surface. These data are derived from the deconvolution of the C_{1s} peak into two

components: the carbons in C-C, C=C and C-H linkages and oxidized carbons such as alcohol, carbonyl, ester, and carboxyl.

X-ray scattering of fibrous materials shows the presence of sharp and diffuse diffraction patterns, indicating crystal phases interspersed with amorphous regions. The concept of the crystalline index¹⁰ is derived from the fact that a portion of the X-ray scattering from a fiber is diffused and contributes to the so-called amorphous background. Thus, a simple method of estimating the crystallinity is obtained by separating the diffraction pattern into crystalline (sharp) and amorphous (diffuse) components. It should be emphasized that the index obtained by the present method is not an absolute numerical result, but rather an indication of relative degree in which some important physical properties are found to correlate.

The crystallinity index (No. 16) is a complicated parameter derived from the X-ray diffraction patterns of amorphous and crystalline reference materials and the sample of interest. A comparison is made between the patterns of standards and the fiber sample of interest as illustrated in Figure 4. The choice of these two standards is rather arbitrary. In this study, highly graphitized carbon black powder (Graphon made by Cabot Co.) was used as the crystalline standard, and the carbon compound with no crystal structure was taken as the amorphous standard (Coke in this case). The index is calculated and averaged over the entire diffraction range of 4-60° at intervals of 0.4° (140 data points). The detailed computation is reported in literature¹⁰ and presented here, in graphic form, in Figure 5. This index has been widely used in fiber industry for the measurement of fiber strength.

DISCUSSION

The structure of fibrous materials could be described as the arrangement and interdispersion of micro-crystalline phases into amorphous regions in an oriented fashion. This simplified fiber structure is not fully compatible with more recent findings¹¹ that the structure consists of many single crystals, chain folding, lamellar crystal growths, and intermediate crystal objects such as axialites and hedrites, lattice dislocation, pores and voids, and one- and two-dimensional ordering in drawn fibers. However, this new crystal-defect concept, as opposed to the earlier two-phase concept, is nevertheless derived from the crystallinity of fibrous materials.

Even the relative degree of crystallinity is not unambiguously defined and is also subject to experimental procedure and measurements. The physical and mechanical properties of fibers are profoundly dependent on the degree of crystallinity, regardless of the chosen method of measurement. The tensile strength and modulus of elasticity of carbon fibers are directly related to the degree of alignment and stiffness of molecular chains parallel to the fiber axis and, hence, to the degree of crystallinity. Likewise, the yield stress of a fiber increases with the degree of crystallinity.

The six fiber samples used in the TTCP program were obtained from two sources. Groups A and B are batches from two different processes. The parameters presented

10. WAKELIN, J.H., VIRGIN, H.S., and CRYSTAL, E. *Development and Comparison of Two X-Ray Methods for Determining the Crystallinity of Cotton Cellulose*. J. Appl. Phys., v. 22, 1959, p. 1654.

11. GUIGON, M., OBERLIN, A., and DESARMOT, G. *Microtexture and Structure of Some High Tensile Strength PAN Base Carbon Fibers*. Fiber Sci. and Technol., v. 20, 1984, p. 177.

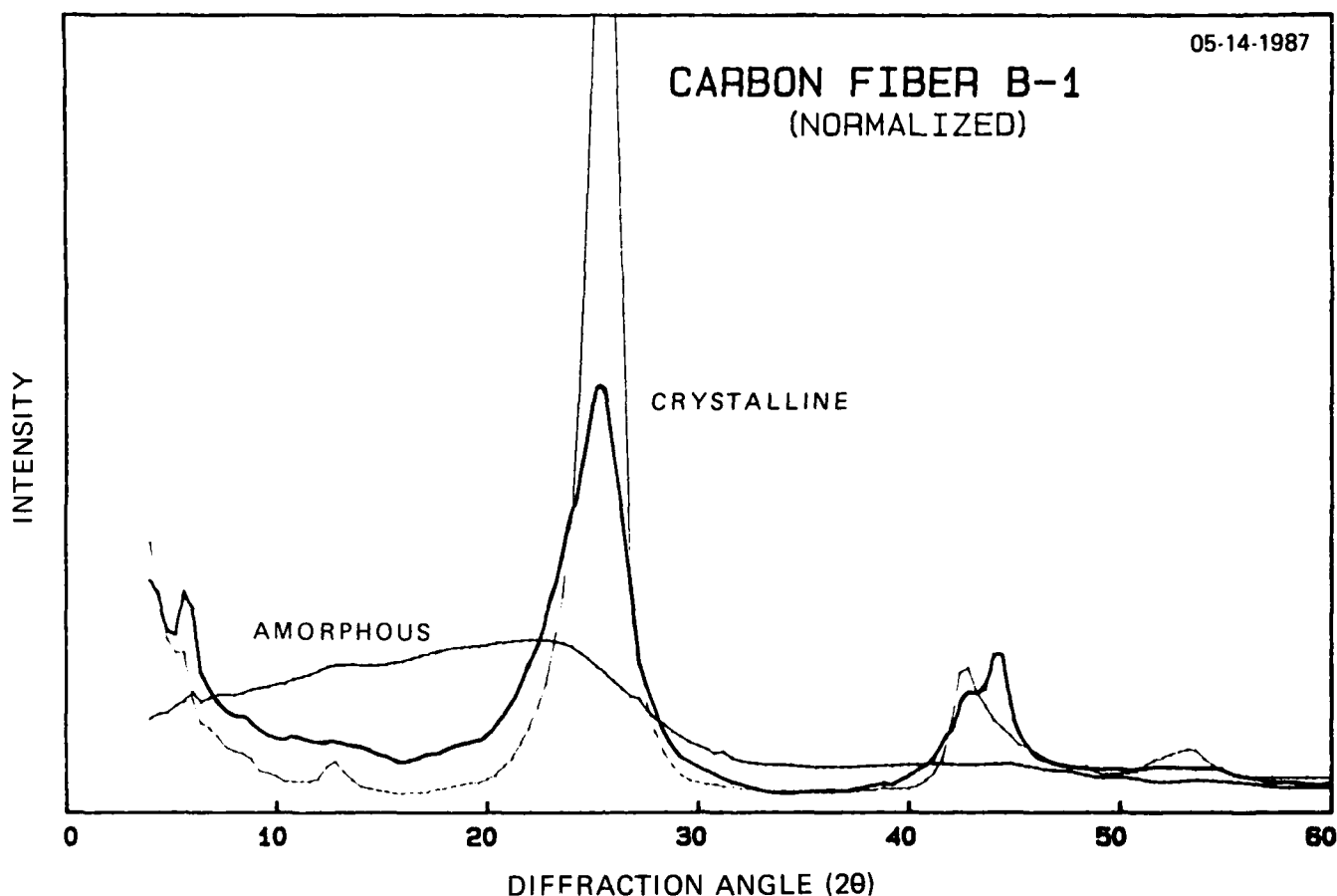


Figure 4. X-ray diffraction patterns of sample crystalline and amorphous reference carbons. These three patterns are compared and integrated to yield crystalline index.

in Table 1 could be used to evaluate/compare the wide range of characteristics of commercial fibers. The differences are clearly shown in the two structure parameters (Nos. 8 and 9) that give the average dimensions of crystallites along the *a*- and *c*-axes. The large crystallite dimensions seem to improve the modulus (No. 2) but decrease the strength (No. 1). Group B has an average of seven basal planes of crystallites as compared to five in Group A. The *d*-spacing (No. 7) is easily obtainable from the X-ray measurement. For a perfect graphite structure, the interplanar spacing is 3.35 Å; an increase in this parameter signifies the extent of deviation from the perfect structure. Thus, the structure disorder and mismatch of the graphite basal planes, which tend to reduce the physical strengths of carbon fibers, increase with increasing length of the *d*-002 spacing. Similarly, the numbers of stacking carbon basal planes also reflect on the fractions of the 002 peak area to the total area (No. 10). The larger the crystallite thickness, the higher the magnitude of parameter No. 10.

Samples A-2 and B-2, surface treated fibers, exhibit a three-fold increase in oxygen surface concentration as compared to untreated fibers (see parameter No. 14). Similarly, parameter 15, which describes the amount of oxidized carbon, is also found to increase. From the peak profiles shown in Figure 3, the peak widths of the O_{1s} peaks after treatment (A-2, B-2) increase markedly, indicating that there are many different forms of oxygen on the surface. An increment of surface oxygen

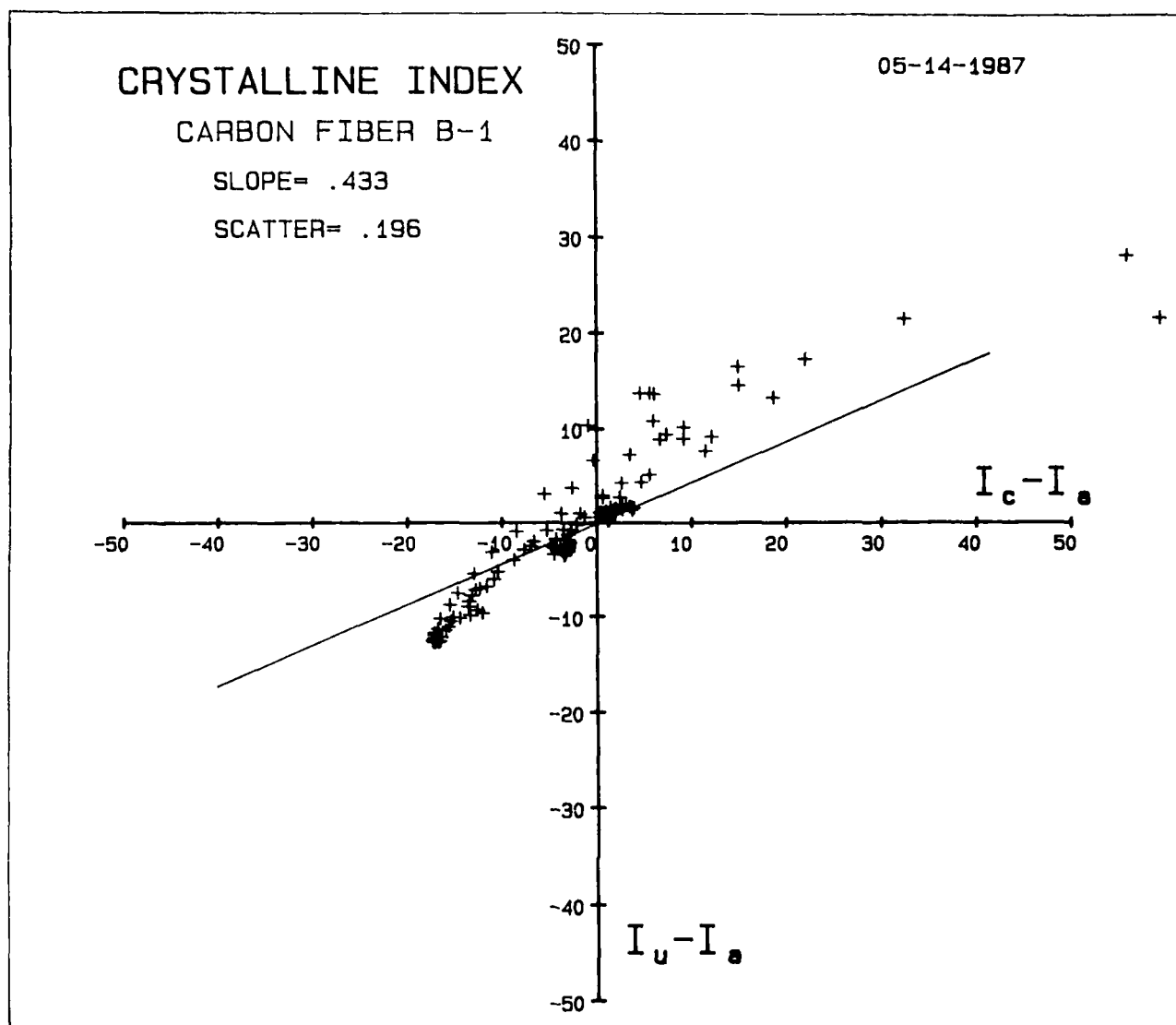


Figure 5. Graphic presentation of crystalline index. Crystalline index is obtained from the $I_c - I_a$ vs. $I_u - I_a$ plot for all diffraction angles from $4-60^\circ$ at an interval of 0.4° . The least-square treatment of data yields the slope as crystalline index and the standard deviation as scatter. Subscripts a, c and u refer to amorphous, crystalline and unknown patterns, and I is a relative diffraction intensity.

magnitudes is also observed on the sized fibers, A-3 and B-3. The oxygen peak positions are found to shift considerably from those of the untreated and treated fibers.

The chemical composition of the sizing compound has more influence over the surface oxygen concentration. The sizing was usually employed less than 1 wt% and was coated on the fiber surface to facilitate handling of bundled filaments. The oxygen magnitudes of parameters 14 and 15 for the sized fibers increased, and are comparable to those of the treated fibers. Evidently the sizing compounds are composed of many oxygen-containing resins. Especially for the sized B-3 fiber, the surface oxygen concentration increased more than that of the treated one. The sizing compound used in the B-fiber is probably different from that used in the

A-fiber. This difference is shown in the carbon peak profiles (Figure 3) having more oxygen functional groups.

Since sizing has little effect on fiber matrix adhesion,¹² the two parameters obtained from the ESCA study might not yield direct information about surface adhesive behavior. Thus, the present surface parameters have no significant meaning. In addition to oxygen, a small concentration of N, presumably derived from the precursor of PAN fibers, is observed on these fibers. The N amount is usually very small; consequently, it is believed to have no significant effect on adhesion.

The crystallinity index¹⁰ is calculated from the complex computation of 140 data sets, giving a least-square regression line in which the slope is the crystallinity index. The standard deviation is rather scattered. This large deviation is partially caused by the misalignment of graphite basal planes in turbostratic graphitic structures and the improper choice of standard materials. The trend of the index is roughly in parallel to the crystalline peak fractions (Nos. 10-12) between the A and B fibers. Since the large scattering is frequently associated with the index, the usefulness of the index in the characterization of carbon fiber requires further examination.

ACKNOWLEDGMENT

The authors wish to acknowledge Dr. G.L. Hagnauer, OML, for his valuable discussion and his encouragement to initiate carbon fiber studies. All samples from TTCP were obtained through his courtesy.

12. HAMMER, G.F., and DRAZEL, I.T. *Graphite Fiber Surface Analysis by X-Ray Photo Spectroscopy and Polar Dispersive Free Energy Analysis*. Appl. Surf. Sci., v. 4, 1980, p. 340.

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Six commercial carbon fibers are characterized by X-ray diffraction, scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS), or Electron Spectroscopy for Chemical Analysis (ESCA). Physical, structural, and surface parameters required to describe major properties of carbon fibers are examined and discussed. The correlation of these parameters is essential for developing optimizing and desired carbon composite materials.

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